भारतीय मानक
Indian Standard

IS 6213 (Part 11) : 2020
ISO 776 : 2011

लुगदी के लिए परीक्षण पद्धति

भाग 11 अम्ल-अघुलनशील भस्म का निर्धारण

(पहला पुनरीक्षण)

Methods of Test for Pulp

Part 11 Determination of Acid-Insoluble Ash

(First Revision)

ICS 85.040

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Paper and its Products Sectional Committee, CHD 15

NATIONAL FOREWORD

This Indian Standard (Part 11) (First Revision) which is identical with ISO 776 : 2011 'Pulps — Determination of acid-insoluble ash' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on recommendation of the Paper and its Products Sectional Committee and approval of the Chemical Division Council.

This standard was originally published in 1975 and was based on ISO 776 'Pulps — Determination of acid insoluble ash', issued by the International organization for Standardization. Now the committee decided to further revise the standard by adopting latest version of ISO 776 on dual number basis.

Acid-insoluble ash primarily consists of silica and silicates. These materials are abrasive in nature and has deleterious effect on the paper making machinery thereby, it is essential to know its content in the pulp before it is fed to the paper making machine. The level of silica in pulp can vary significantly depending on several factors, including wood type, water quality, and the presence of silicate based additives, such as clay and talc. For example, in bleached kraft pulps, the silica mass fraction can range from under 100 mg/kg to over 1 000 mg/kg.

The text of the ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain terminologies and conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker, while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their respective places, are listed below along with their degree of equivalence for the editions indicated.

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 638 Paper, board and pulps — Determination of dry matter content — Oven drying method	IS 6213 (Part 21) : 2019/ISO 638 : 2008 Methods of test for pulp: Part 21 Paper, board and pulps — Determination of dry matter content — Oven-drying method (*first revision*)	Identical with ISO 638 : 2008
ISO 1762 Paper, board and pulps — Determination of residue (ash) on ignition at 525°C	IS 1060 (Part 4/Sec 2) : 2018/ISO 1762 : 2015 Methods of sampling and test for paper and allied products: Part 4 Methods of test for paper, board and pulps, Section 2 Determination of residue (ash) on ignition at 525°C	Identical

The technical committee has reviewed the provisions of the following International Standard referred in this adopted standard and has decided that it is acceptable for use in conjunction with this standard:

International Standard	*Title*
ISO 7213	Pulps — Sampling for testing

(*Continued on third cover*)

Indian Standard

METHODS OF TEST FOR PULP

PART 11 DETERMINATION OF ACID-INSOLUBLE ASH

(First Revision)

1 Scope

This International Standard specifies a method for the determination of the acid-insoluble ash of pulp. It is applicable to all types of pulps.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

ISO 1762, *Paper, board and pulps — Determination of residue (ash) on ignition at 525 °C*

ISO 7213, *Pulps — Sampling for testing*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
acid-insoluble ash
insoluble residue remaining after a pulp sample is ignited in a furnace at 525 °C ± 25 °C and the ash treated with hydrochloric acid as specified in this International Standard

4 Principle

A test specimen is weighed in a heat-resistant container, ignited in a muffle furnace at 525 °C ± 25 °C, and the residue is treated with hydrochloric acid. The insoluble residue after acid treatment is filtered, washed, ignited at 525 °C and weighed. The moisture content of a separate test portion is also measured. The acid-insoluble ash is then determined, in milligrams per kilogram, on an oven-dry basis, from the mass of the insoluble residue after ignition and the dry matter content of the sample.

5 Reagent

Use only analytical grade reagent and distilled water or water of equivalent purity.

5.1 Hydrochloric acid, 6 mol/l solution.

Carefully, in a fume hood, dilute 500 ml of hydrochloric acid (density at 20 °C = 1,19 kg/l) to 1 000 ml with water.

6 Apparatus

6.1 Heat-resistant evaporating dishes, of capacity 50 ml to 100 ml, and **crucibles** with a capacity of 20 ml. Platinum containers are preferred, but porcelain or silica may be used.

6.2 Steam bath or **electric hotplate**, capable of maintaining a surface temperature of 110 °C to 130 °C.

6.3 Analytical balance, of capacity 100 g, accurate to 0,1 mg.

6.4 Desiccator.

6.5 Muffle furnace, capable of maintaining a temperature of 525 °C ± 25 °C.

6.6 Filter paper, quantitative, ashless, double acid-washed, of a type recommended for fine precipitates (e.g. Whatman No. 42[1)] or equivalent).

6.7 Drying oven, capable of maintaining an air temperature of 105 °C ± 2 °C and suitably ventilated.

7 Sampling and preparation of test specimen

If the analysis is done in order to evaluate a pulp lot, obtain a representative pulp sample as described in ISO 7213. If it is not, report the source of the sample and, if possible, the sampling procedure used. Sufficient sample shall be collected to allow for at least duplicate determinations and for the determination of moisture content.

Obtain a test specimen consisting of small pieces, not larger than 1 cm^2, from various parts of the sample in such a manner as to be thoroughly representative of it. The mass of the test portion shall be such that the mass of the acid-insoluble residue is greater than 10 mg. A test portion weighing 25 g is sufficient in most cases.

NOTE In samples with very low levels of acid-insoluble residue, it might be necessary to obtain a larger test specimen and to ignite it in more than one portion.

8 Procedure

Carry out the procedure in duplicate.

Condition the test specimens and moisture specimens in the laboratory atmosphere until they reach equilibrium moisture.

Determine the moisture content of the sample as described in ISO 638. Weigh this test portion at the same time as the test portion used for ash determination.

Ash the test specimens as described in ISO 1762, in a dish previously ignited in the muffle furnace, to a constant mass (to the nearest 0,1 mg). If the dish is too small to hold the entire specimen, the specimen shall be ashed in portions. Place a portion of the specimen in the dish and burn it gently to avoid any loss. Then add the next portion to the dish and burn it gently. Continue this process until the entire specimen has been carbonized, as indicated by the absence of black carbon particles.

Cool the dish to room temperature in a fume hood, add 5 ml of 6 mol/l HCl (5.1), and evaporate carefully to dryness on a steam bath or hotplate (6.2). Repeat the treatment. Add a third 5 ml aliquot of 6 mol/l HCl (5.1) to the residue, heat for 2 min on the steam bath or hotplate, and dilute with 20 ml of water.

Quantitatively transfer the residue by filtering through the filter paper (6.6), being careful to remove all the residue from the dish with water. Wash the residue several times with hot water until the filtrate is free of chloride ions.

The absence of chloride ions in the filtrate may be confirmed with a few drops of 5 % silver nitrate. The filtrate should not produce a white precipitate or opalescence.

Carefully place the filter paper and residue into a tared 20 ml crucible or heat-resistant evaporating dish (6.1). Heat gently until dry, then ignite at 525 °C ± 25 °C until the entire specimen has been ashed, as indicated by the absence of black carbon particles. Cool the crucible in a desiccator and weigh to the nearest 0,1 mg. Repeat the ignition until a constant mass is obtained.

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1) Whatman No. 42 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

9 Calculation

Calculate the mass fraction of acid-insoluble ash according to Equation (1):

$$w_a = 1\,000 \times \frac{m_1}{m_2} \tag{1}$$

where

w_a is the mass fraction of acid-insoluble ash, expressed as milligrams per kilogram of the oven-dry sample;

m_1 is the mass of insoluble residue after ignition at 525 °C ± 25 °C, in milligrams;

m_2 is the mass of the sample, on an oven-dry basis, in grams.

Report the mean of duplicate determinations to the nearest 5 mg/kg.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard (ISO 776:2011);

b) the date and place of testing;

c) a complete identification of the sample tested;

d) the result, expressed as indicated in Clause 9;

e) any departure from the procedure described in this International Standard or any other circumstances which may have affected the result.

Annex A
(informative)

Precision

A.1 General

In August 2009, an international round robin was performed in which eight laboratories from five different countries participated.

Three samples of different types of pulp were tested. Each sample was disintegrated, to ensure its homogeneity, and made into handsheets. Several handsheets from each sample were submitted to the participating laboratories for testing according to this International Standard. For the two hardwood bleached kraft pulp samples, the data from two of the eight laboratories were considered as outliers and were not included in the precision statement.

The calculations were made according to ISO/TR 24498, *Paper, board and pulps — Estimation of uncertainty for test methods*.

The repeatability and reproducibility limits reported are estimates of the maximum difference which should be expected in 19 of 20 instances, when comparing two test results for material similar to those described under similar test conditions. These estimates may not be valid for different materials or different test conditions.

NOTE Repeatability and reproducibility limits are calculated by multiplying the repeatability and reproducibility standard deviations by 2,77, where $2{,}77 = 1{,}96 \times \sqrt{2}$.

A.2 Repeatability

Table A.1 — Estimation of the repeatability of the test method

Sample	**Number of laboratories**	**Mean value** mg/kg	**Standard deviation** s_r mg/kg	**Coefficient of variation** $C_{V,r}$ %	**Repeatability limit** r mg/kg
Hardwood bleached kraft pulp (low ash)	6	1 368	72,6	5,3	201
Hardwood bleached kraft pulp (high ash)	6	2 909	72,4	2,5	201
Clay-filled mechanical pulp furnish	8	112 955	4 611	4,1	12 781

A.3 Reproducibility

Table A.2 — Estimation of the reproducibility of the test method

Sample	Number of laboratories	Mean value mg/kg	Standard deviation s_R mg/kg	Coefficient of variation $C_{V,R}$ %	Reproducibility limit R mg/kg
Hardwood bleached kraft pulp (low ash)	6	1 368	112	8,2	311
Hardwood bleached kraft pulp (high ash)	6	2 909	165	5,7	458

(*Continued from second cover*)

In this adopted standard, reference appears to certain International Standards where the standard atmospheric conditions to be observed are stipulated which are not applicable to tropical / sub-tropical countries. The applicable standard atmospheric conditions for Indian conditions are 27 ± 2°C and 65 ± 5 percent relative humidity and shall be observed while using this standard.

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

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